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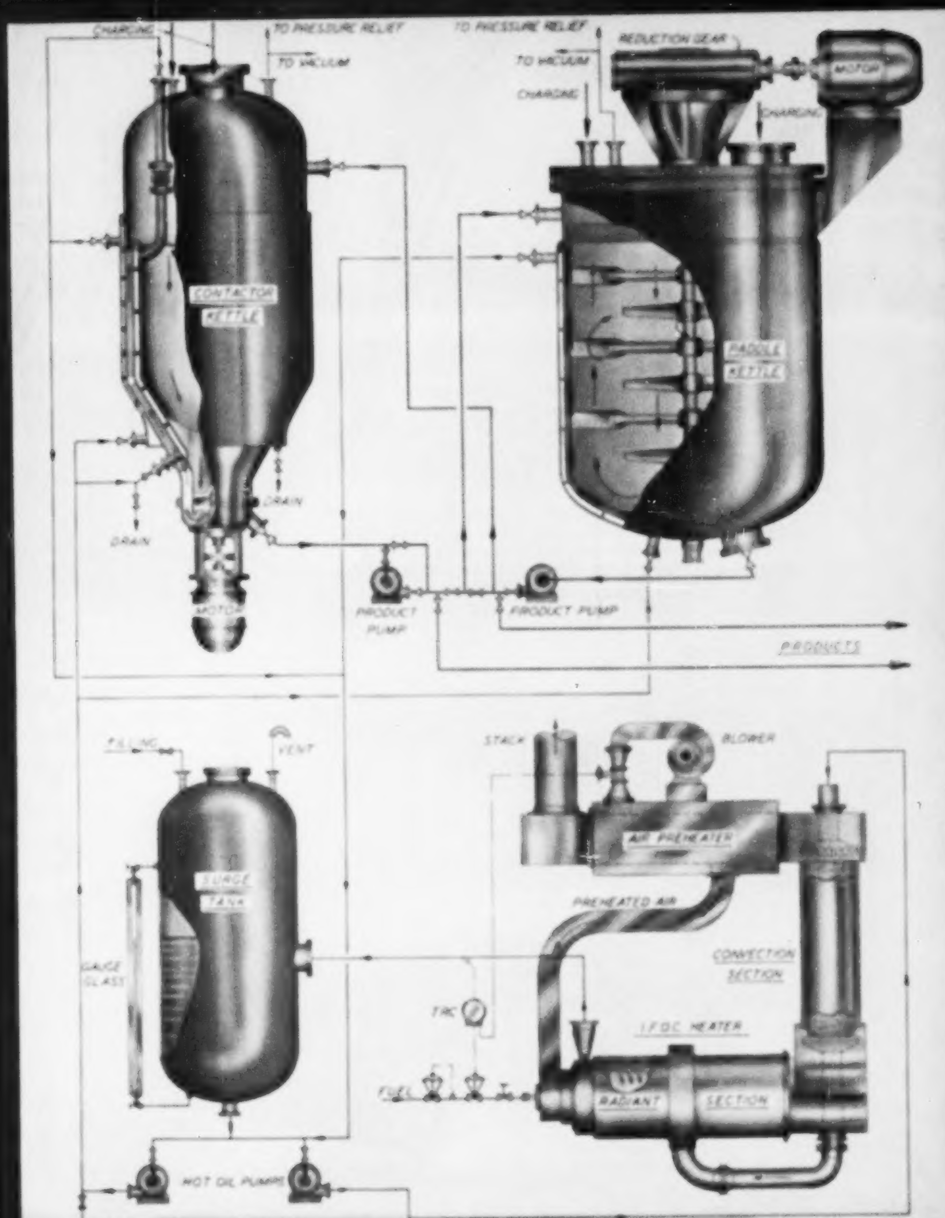

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NATIONAL LUBRICATING GREASE INST.
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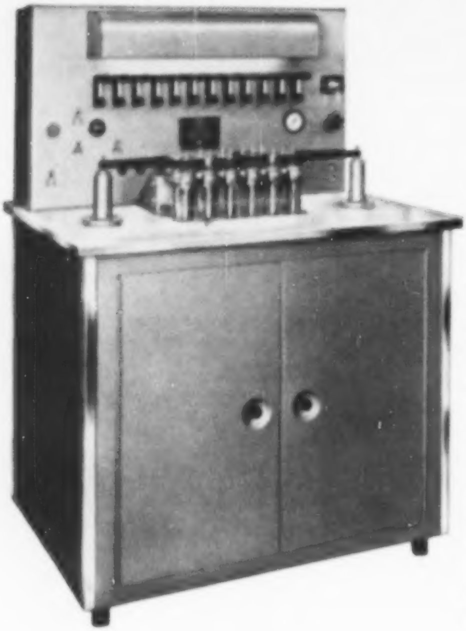

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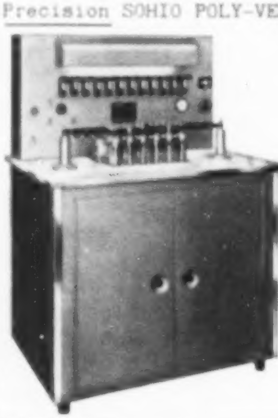

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The **INSTITUTE SPOKESMAN**

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In This Issue

JULY, 1950

Volume XIV Number 4

ABOUT THE COVER

The front cover this month shows in Flow Sheet form the Stratco Grease Making Process and the apparatus required therefor. The three basic items of equipment required are the Stratco Contactor and Oil Heating System and an efficient double stirrer paddle kettle arranged for oil heating.

The process is one in which batches of all types of greases can be made with precision in extremely short time cycles, for example: calcium greases in 60-75 minutes and soda greases in 120-135 minutes. Batches are uniform and easily duplicated. Grease making is thus converted from an art to a science of precision. The short time cycles make possible manufacture during normal forty hour week operation eliminating the difficulties of shift work and supervision.

The equipment used, especially the Stratco Contactor, is very flexible and can be used for many diversified uses, such as sulfurizing oil, manufacture of E. P. and other fluid greases and all miscellaneous soaps.

By application of this equipment and process, existing obsolete grease plants can be converted to this modern system substantially reducing fat consumption, operating personnel and hours of operation, thereby materially reducing costs.

President's page

by Arthur J. Daniel, President, N.L.G.I.

SERVICE — KEY TO PAST AND FUTURE

The rapid growth and expansion of the NLGI is visible proof that the organization has served its members well. Through the unselfish work of our various committees and members, the technical development and application phases of the Institute's work has been outstandingly successful. Service, the key to our past success, is also the key to our future. Today, the NLGI has before it the task of not only continuing its past work but of finding additional ways in which its value to the individual member can be increased.

RESEARCH — BACKBONE OF PROGRESS

Technical research has been and will always be the backbone of progress, but we must not become so absorbed in the alignment of molecules or mechanical processes that we consider achievement in this field to be the end result of our efforts. The true value of research lies in its ability to increase the welfare of our fellow men. Stated simply, we can increase scientific knowledge and create "better lubricants", but it will have little meaning unless we can spread this knowledge to the proper source . . . the lubricating grease engineer and applicator must use our discoveries in their work.

Progress in this direction has been accomplished through cooperative committees with other industries and the technical knowleage we have gained has been most valuable in this work. Here is the type of action that can produce tangible results for our industry.

APPLYING RESEARCH

The problem of wider dissemination of knowledge outside the confines of our group implies the use of a public education program. However, the financial burden of such a program, if carried to a successful conclusion, would probably be impractical at this time. Where then can we turn to spread the benefits of our work?

I firmly believe that the answer lies in "human research". While we can continue to concentrate on the technical aspects of our industry's problems, we can also inquire into the lubrication and purchasing habits of people who use the products of our industry. The existing data on this subject are wholely inadequate.

EXPANDED SERVICE TO MEMBERS

To place these thoughts in the form of a concrete proposal, I would suggest that the NLGI embark upon a program to study and gather information of value to the marketing as well as the technical side of our industry. The dissemination of such information either through the "Spokesman" or in bulletin form would be of inestimable value to the membership of our organization. However, I well realize that such an increase in the scope of the NLGI's activities is a matter in which all members should have a voice.

The expansion of needed service to its members is a healthy, normal sign of growth for any organization. The NLGI has an enviable record for service and if we can supplement its present activities with further aid and assistance in the procurement and dissemination of marketing data, it would be a forward step, indeed.

by T. D. SMITH,
E. AMOTT, and
L. W. McLENNAN

Union Oil Company
of California
Oleum, California

The Syneresis of LUBRICATING OIL GREASES

"A number of tests have been devised to measure this property in a matter of hours or days. This paper describes four such tests, and some of the factors involved in the syneresis of grease."

INTRODUCTION

Thomas Graham (14) observed in 1861, that when a freshly prepared gel of silicic acid is allowed to stand, it spontaneously exudes liquid. To this phenomenon he applied the term "syneresis". It is of very general occurrence, having been observed with inorganic gels, such as vanadium pentoxide (42) and silver nitrate (25), organic gels, such as dyes, (21,23), rubber (39), starch (6), albumen (16), viscose (22,29), agar (35), and gelatin (1,18,22); and in a wide variety of technical products, including glue (32), margarine (7), and bread (30).

As judged from wide experience with experimental and commercial lubricating greases, syneresis in some degree can be regarded as a normal expectation, rather than the contrary, particularly for greases which are of N.L.G.I. 2 grade or softer, and which have been stored for a prolonged period. Many specifications recognize this by placing a limitation upon the amount of free oil to be tolerated. It would, then, be useful to have some means of predicting to what extent a grease will separate oil in storage. A number of tests have been devised to measure this property in a matter of hours or days. This paper describes four such tests, and some of the factors involved in the syneresis of grease.

THE CRATER TEST

This is simply a storage test, conducted by placing a sample of grease in a 3-ounce can, of diameter 2-1/8 inches and height 1-3/8 inches, with a 1/8" diameter hole in the center of the bottom. The surface of the grease is shaped into a cone, the apex being the hole in the bottom of the can, and the base being the rim of the can. This can is placed on a small stand in a larger can, so that exuded oil may run out, and the assembly is then stored in a constant temperature box maintained at a pre-determined temperature. The loss in weight is determined after known periods of time.

In many cases, when a temperature of 150° F. was employed, the surface of the grease collapsed, so that exuded oil could not flow from the can. On the other hand, in order to obtain measurable results when the test was carried out at 100° F., it was necessary to considerably increase the time of storage. Therefore, most of the tests were carried out at 130° F. The results were expressed as weight percent oil loss after 200 hours. When carried out at 100° F., the period was doubled.

If bleeding occurred to any appreciable extent, the initial rate of syneresis was relatively large, but decreased rapidly with time. In most cases it was found that if the logarithm of the oil loss (L) was plotted against the logarithm of time (T), a straight line was produced; i.e. the following relation was valid for the length of time investigated:

$$L = kT^{s}$$

k = constant

s = slope of log L vs. log T.

Crater Test results for certain greases have satisfied this relation for storage time of up to 5,000 hours for the 100° F. test and up to 3,000 hours for the 130° test. Fig. 1 illustrates typical results, and includes an anomalous cuurve which is discussed below. Interpolation from the log-arithmic plots was a convenient method of obtaining the oil loss at 200 hours. The value of s, although not directly proportional to the rate of syneresis, may be used as an indication of the shape of the loss vs. time curve.

The above relation would not be satisfied for an indefinite length of time. However, only one definite case of deviation (Fig. 1) was observed in a period of less than 1,000 hours, which was not due to collapse of the grease surface causing the outlet hole to become plugged. In this one case it was found that, after the point of failure of the logarithmic relationship, the equation of Farrington and Humphreys (see below) was satisfied. The explanation for this occurrence is not immediately obvious, and, being an isolated case, it was not further investigated.

It is not likely that any one test could predict the exact behavior of a grease on storage, as there will be a number of environmental variables which might be encountered, which it would be too time consuming to consider in every case. However, an examination of greases stored in different

localities exhibiting various climatic conditions, indicated that the Crater Test gave a satisfactory measure of how a grease would behave under not too extreme conditions. (Tables IA and IB). This method was therefore taken as a standard measure of storage stability throughout the following investigations. It is simple to perform and is believed to have a wider range than other storage tests. (e.g. AN-G-3a Test, Cone Bleeding Test, Basket Test, etc.) (13,33).

THE CENTRIFUGE TEST

The application of centrifugal force to accelerate the separation of oil from greases has been described by Folda (12) and others. However, it is believed that no detailed information on the reliability of the test has been published.

In the present study, samples of greases were weighed into 50 ml. tubes and centrifuged for 24 hours in an International clinical centrifuge. The speed of the centrifuge was maintained at 2,500 ± 20 rpm., which was equivalent to a relative centrifugal force of approximately 750. The oil which separated was decanted and weighed, the final result being expressed as percentage loss by weight of the original grease.

The greases listed in Table II differed only in the viscosity of the oil component. It will be observed that whereas the storage stability, as measured by the Crater Test, was approximately constant, the amount of oil separated by centrifugation showed a pronounced dependence on the viscosity of the oil component. In confirmation of the Crater Test results, it was found that the same amount of oil separated from 35-lb. samples of these three greases after standing in the laboratory for three months.

A series of greases was tested, and the Centrifuge Test results could not be correlated with the Crater Test results (Table III). It was observed in most cases that oil separation, as measured by the 130° Crater Test, occurred to a greater extent after, than before, centrifugation. The following explanation is suggested. Centrifugal force acting on the grease structure would tend to collapse the capillary network. As oil migrated along the tubes, a flow which would be dependent on the oil viscosity, small quantities of oil could collect in minute vacuoles within the main anastomotic structure. Such a structure would conceivably be less stable, and consequently be more syneretic, than the original structure.

THE PRESSURE FILTRATION TEST

The technique employed in this study was a modification of that devised by Roehner and Robinson (35). Grease was pumped into a pressure filtration funnel (Fig. 2) containing a fine fritted glass plate, ensuring that no air cavities were included. The funnel was connected to a burette containing oil of the same type as the oil component of the grease. The apparatus was maintained at 110° F., and a measured constant pressure supplied to the grease by means of compressed air. Burette readings were taken at frequent intervals to determine the volume of oil being filtered from the grease.

Graphs of oil loss against time, plotted on log-log paper, were straight lines, the slopes of which were all equal or very nearly equal to ½. It was found that this behavior satisfied Poiseuille's equation, although this relationship refers to an ideal capillary tube. Poiseuille's equation states:

$$I = \frac{PR^4T}{8 \eta l}$$

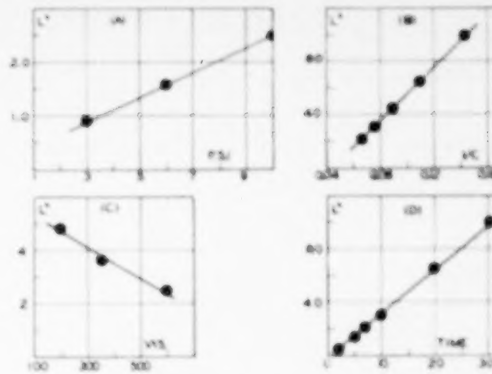


FIGURE 1. SYNERESIS IN CRATER TEST VS. TIME
ORDINATE: Percent Loss in Weight
ABSCISSA: Time in Hours

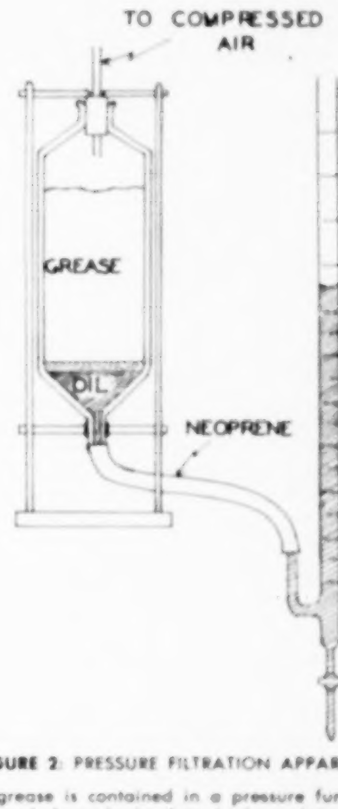


FIGURE 2: PRESSURE FILTRATION APPARATUS.

The grease is contained in a pressure funnel. Oil is forced through the fritted glass plate, the increase in volume being measured in the burette. The apparatus is maintained at constant temperature.

where L = volume of liquid passing through capillary
P = pressure
R = capillary radius
l = capillary length
T = time
n = viscosity of liquid

In the pressure filtration apparatus the capillary length (l) may be taken as the thickness of the accumulated filtering bed. This is directly proportional to L, the amount of oil separated, and to C, the concentration of soap in the grease.

i.e. $l = kLC$, where k is a constant.

Substituting in Poiseuille's equation $L^2 = \frac{k'PR^4T}{C\eta}$

This equation was tested in the following manner: Six different greases were examined in the pressure filtration apparatus, results being obtained on each grease for three different pressures. It was found that the square of the oil loss at time T (L^2), was directly proportional to the applied pressure. Similarly it was found that L^2 was inversely proportional to the soap content. In this case, one grease was divided into several portions, each of which was diluted to a different extent with oil to provide a series of greases differing only in soap content.

It was also found that L^2 was inversely proportional to the viscosity of the oil component using any one type of grease. These ratios are illustrated in Fig. 3. Rather surprisingly, the value of R remains constant, although it might be anticipated that this term would vary with time. However, the usual physical significance of Poiseuille's equation should not be assumed completely in the present case.

The above behavior might suggest that the pressure filtration apparatus could not be used to predict storage stability. A comparison of the results of this test with those of the Crater Test (Table III), indicated that no correlation existed. Fibrous greases appeared to lose oil more readily under pressure with filtration than greases exhibiting a buttery texture.

Roehner and Robinson suggested that this test might be useful to predict the separability characteristics of greases contained in pressure devices where filtration could occur.

THE HERSCHEL PRESS TEST

A detailed account of the Herschel press has been given by Farrington and Humphreys (10). The application of a similar type of press to determine the stability of sodium stearate gels has been described by Doscher and Vold (8). Briefly, the method consisted in placing a weighed sample of grease (about 2 grams) between two filter papers, which were subsequently placed between two pieces of blotting paper and clamped into a device in which a heavy plunger could be inserted. The apparatus was maintained at 100° F., and a 5-lb. plunger employed to apply a pressure of 1.81 psi to the grease sample. By weighing the filter paper "sandwich" at frequent intervals the amount of oil loss was obtained.

TABLE 1A
COMPARISON OF FIELD DATA WITH CRATER TEST RESULTS

		Ounces of Oil in ½ bbl. Containers[1]		
Grease[2]	Crater Test 100° F., 400 hrs.	Location A	Location B	Location C
B1	0.00	1	0	2
B2	0.00	2-3	2	4
B3	0.35	3	2	4
B4	1.05	10	16	4

1. Stored in locations exhibiting high day and low night temperatures. The surface condition varied considerably, grease having been removed from some of the containers.
2. Data on these greases is given in Table VI.

TABLE 1B
COMPARISON OF SEPARATION IN STORAGE WITH CRATER TEST RESULTS

Grease[2]	Crater Test 130° F., 200 hrs.	Volume of Oil[1], ml.	Time of Storage in Months
B5	0.00	trace	10
B2	0.05	10	10
B6	0.10	20	7
B7	0.10	40	5
B8	0.20	10	5
B9	0.25	30	10
B10	0.35	70	10
B11	0.35	80	5
B3	0.50	100	8
B12	1.00	180	10

1. The volume of oil which had collected on the surface of 35-lb. pail samples of the greases, after storing in a warehouse. Although the temperature effects were similar for these samples, the time of storage varied from 5-10 months, and the surface conditions varied

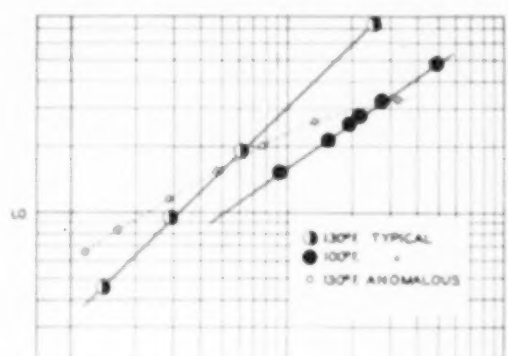


FIGURE 3

RELATIONSHIPS SATISFIED IN THE PRESSURE FILTRATION APPARATUS

A. (Percent loss in weight)[2] at constant time vs. applied pressure (pounds per square inch).
B. (Percent loss in weight)[2] at constant time vs. the reciprocal of soap content.
C. (Percent loss in weight)[2] at constant time vs. the viscosity of the oil component, (S.U.S. at 100 degrees F.).
D. (Percent loss in weight)[2] of one grease vs. time in hours.

considerably, all the containers having been broken into to some extent.

2. Data on these greases is given in Table VI.

Farrington and Humphreys found that their results satisfied the empirical equation:

$$\frac{T}{L} = a + bT$$

where T = time

L = oil loss

a and b are constants

Therefore, a plot of $\frac{T}{L}$ against T should yield a straight line.

This behavior was confirmed in the present studies (Fig. 4). The initial deviation was also observed by Farrington and Humphreys, who believed that it was due to the time required to wet the filter paper with oil.

From the above equation it will be seen that when $T = O$, $\frac{dL}{dT} = \frac{1}{a}$, and is referred to as the "initial rate of loss"; also, when $T = \infty$, $L = \frac{1}{b}$, and is referred to as the "ultimate loss".

The values of a,b, and the oil loss at T = 0.01 hours, for a number of greases, are given in Tables II and III. None of these values could be correlated with the Crater Test results. It was concluded that this test cannot be used as a means of predicting oil separation in storage.

Farrington and Humphreys found that with two cup greases differing only in the viscosity of the oil component, the ultimate loss was the same in each case, although the initial rate of loss was higher for the grease containing the lighter oil. This behavior was confirmed using three barium greases (Table II).

TABLE II

TEST RESULTS ON BARIUM GREASES CONTAINING OIL COMPONENTS OF VARYING VISCOSITY

Grease	Viscosity of Oil Component S.U.S. 100°F.	130° Crater Test: Before Centrifugation Loss[1]	S[2]	After Centrifugation Loss[1]	S[2]	Wt. Percent loss by Centrifugation	Pressure Stability Oil Loss[3]	S[2]	Herschel Press a	b	Loss when T = 0.01 hrs.
B13	600	0.35	2.05	0.80	0.41	1.20	0.51	0.51	0.008	0.028	1.2
B14	600	0.10	0.70	—	—	1.15	—	—	—	—	—
B15	350	0.35	0.95	0.50	0.23	2.45	0.60	0.49	0.006	0.028	1.6
B16	200	0.35	0.84	0.50	0.22	5.10	0.70	0.47	0.004	0.027	2.4
B17	200	0.35	0.97	0.40	0.19	6.00	—	—	—	—	—

1. Weight percent after 200 hours storage.
2. Slope of log loss vs. log time.
3. ml/100g. after 1 hour.

See Table VI.

TABLE III

OIL SEPARATION TEST RESULTS

Grease[5]		130° Crater Test Loss[1]	S[2]	Pressure Stability[4] Oil Loss[3]	S[2]	Centrifuge Wt. % Loss	Herschel Press a	b	Wt. Percent Loss when T = 0.01 hrs.
Sodium base									
grease	S1	0.10	0.47	1.00	0.51	0.00	0.020	0.019	0.5
	S2	0.50	0.91	1.25	0.55	0.45	0.001	0.022	8.2
Cup grease									
	C1	0.25	0.85	1.05	0.51	0.05	0.003	0.027	3.1
	C2	—	—	—	—	—	0.008	0.039	1.2
Barium grease									
fibrous	B18	2.40	0.71	1.55	0.55	6.30	0.002	0.019	4.6
	B19	0.05	—	1.00	0.49	—	0.013	0.021	0.8
buttery	B20	1.00	0.71	0.48	0.51	1.55	0.011	0.027	0.9
	B21	0.10	—	0.33	0.51	0.00	0.028	0.033	0.4
	B2	0.05	0.47	0.58	0.51	0.75	0.018	0.027	0.6
	B6	0.10	0.54	0.70	0.49	4.10	0.007	0.028	1.4

1. Weight percent after 200 hours storage.
2. Slope of log loss vs. log time.
3. ml/100g. after 1 hour.
4. Pressure of 10 psi. employed.
5. See Table VI.

FACTORS AFFECTING SYNERESIS

Influence of Temperature

The rate and extent of syneresis from greases is increased by raising the temperature. This has been observed by many workers, and has been confirmed in these studies. Almost all syneretic gels exhibit this behavior (18), although it has been reported that syneresis from sodium oleate-pinene gels (31) is increased by decrease in temperature.

If the temperature is raised beyond certain limits, it is possible that phase transitions may occur leading to the formation of unstable structures very different from those existing at room temperature (41). Caution should therefore be exercised in determining suitable temperatures at which to conduct storage tests, such as the Crater Test previously described. In evaluating the results of such tests it should also be noted that the temperature dependence of syneresis may vary widely for different greases. Thus two greases which show a similar oil separation at 100° F. may show a markedly different separation at 150° F.

A number of Crater Tests were carried out at a series of temperatures, on greases of varying soap content. In many cases the following relationship was satisfied:

$$\log L = a + b\,\theta$$

where L = oil loss, weight percent

θ = temperature

a and b are constants, depending on the nature of the grease.

The intercept, a, on the log L axis, which is a measure of the amount of oil separation, was greater the lower the soap content. However, Figure 5 illustrates that with grease of different soap content made from the same soap concentrate, the slope, b, was about the same in each case. In other words, the temperature dependence of syneresis is independent of soap concentration, within the range studied, for any one grease, although the magnitude of syneresis is greater the lower the soap content. The temperature dependence is an important factor when considering the effect of environment on greases.

All the storage tests which have been proposed involve a constant temperature. However, packages may be stored in locations which exhibit considerably different day and night temperatures, so that the temperatures of the stored greases may rise and fall between quite wide limits. The prevalence of such conditions justifies further investigations on the effect of the varying temperature on syneresis.

INFLUENCE OF STATIC HEAD

The method of storage may also affect the extent of syneresis to a large extent. Experiments were carried out to determine the effect of variations in syneresis with static head of grease. Such conditions would, for example, be exhibited where the surface of the grease in a container had been gouged, leading to the formation of large depressions. It appears that syneresis is generally increased by this procedure.

A number of rectangular boxes of varying dimensions were filled with grease and stored at 130° F. with the open side verticle. The exposed vertical grease suurface was of constant width, but of varying height. It was found that the weight percent of separated oil increased with increase in the head of grease (Table IV). Under the conditions of these experiments, if increase in head did not affect the amount of syneresis, the weight percent of separated oil would be constant, as the method essentially involved stacking unit cubes of grease on top of each other with no intervening partitions, so that the head was transmitted throughout the grease column. Similar results were obtained for a number of different greases.

INFLUENCE OF SURFACE AREA

The effect of variations in surface area was also investigated. Cylindrical containers of constant height (1 inch) were

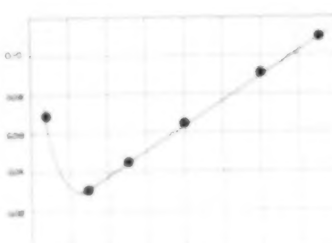


FIGURE 4. (Right) TYPICAL RESULT OBTAINED WITH THE HERSCHEL PRESS.
ORDINATE: Time (hours) / Loss (Weight percent)
ABSCISSA: Time in Hours.

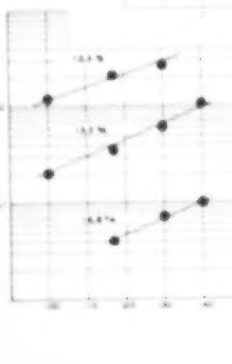


FIGURE 5. (Left) THE EFFECT OF TEMPERATURE ON SYNERESIS.
ORDINATE: Weight per cent oil loss.
ABSCISSA: Temperature, degrees F.

TABLE IV

TYPICAL RESULTS ILLUSTRATING THE EFFECT OF HEAD ON SYNERESIS

Container Dimensions	Area/Volume Ratio, inches⁻¹	Weight Percent Oil Separation after 70 hrs. at 130° F.
1" x 1" x 1"	1	.06
1" x 1" x 2"	1	.22
1" x 1" x 4"	1	.32
1" x 1" x 6"	1	.61
2" x 2" x 2"	½	.25

TABLE V

EFFECT OF SURFACE AREA ON SYNERESIS

Grease	Temperature °F.	Time Hours	Weight Percent Oil Separation ½[1]	2[1]	4[1]
S1	150	20	0.2	0.8	1.5
S2	100	72	0.7	1.0	1.2
C1	130	72	0.3	0.5	0.8
B6	150	48	0.7	1.8	2.2
B2	150	48	0.5	1.2	1.6

1. Curved surface area/volume, inches⁻¹.

constructed, in which the bases were solid, and the walls of 40-mesh gauze. Three different radii (½ inch, 1 inch, and 3 inches) were used so that, although the head of grease was constant, the curved surface area/volume ratio varied in the order 2/3, 2 and 4 reciprocal inches. These containers were filled with grease, stored at slightly elevated temperatures, and the loss in weight determined after suitable intervals of time.

The results given in Table V illustrate that increase in surface area causes a definite increase in the extent of syneresis. This factor must therefore be carefully considered when evaluating or predicting oil separation from greases in packages. It is interesting to note that the syneresis of certain other gels (starch (6), and sodium oleate-pinene (31)) is increased with increasing area. Owing to the difficulties in measuring syneresis, a precise correlation between surface area and separation is not possible. In fact, in the case of a silica gel, contradictory results have been published (11,15).

INFLUENCE OF CONCENTRATION

The quantity of syneretic liquid exuded from gels may be either increased or decreased with increasing concentration of the thickening agent, depending on the nature of the gels. Syneresis is decreased with increase in concentration in the case of dyes, (23), starch (27), cellulose acetate (26), albumen (16) and agar (36). The reverse behavior has been observed with silica gels (15), and rubber-benzol solutions after vulcanization with sulfur monochloride (2). Viscose may behave in either way depending on the pH of the gel (28).

All the reports on grease indicate that syneresis occurs to a greater extent the lower the soap concentration (10,13,35, 40). This has been confirmed in these studies for all the tests employed.

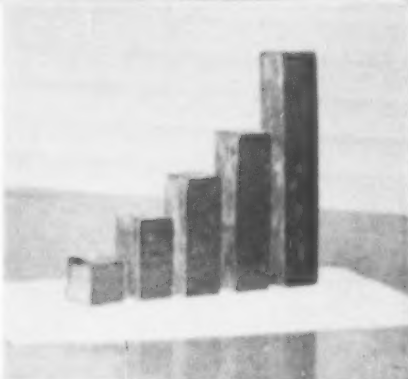
DISCUSSION

It is probable that soap-oil systems exist as duplex (2) gels, in which the oil is contained in two ways, one closely bound to the elementary soap micellae, the remainder being physically immobilized within the capillary spaces between the fibrillar network. Electron microscopy (3,9,28,37) has revealed the soap fibers which constitute the gel framework.

A number of different mechanisms may be involved in the exudation of oil from such a structure. It has been suggested that some separation may occur from the larger capillaries in a grease structure, simply because of the lower static head of liquid which they can contain (4). Growth of soap crystallites may provoke syneresis by collapsing the capillary spaces and reducing the area of the soap surface. The capillaries may also collapse spontaneously due to the presence of internal stresses. Kuhn (18) considered this as a continuation of the disintegrative process of gelatinization, quoting evidence which indicated the presence of appreciable stresses within a number of gels, and observing that such a contention does not involve assumptions concerning the structure of the concentrated phase. It appears unnecessary to assume the existance of lyospheres or solubilization to account for the syneretic behavior of greases. All the available evidence indicates that the exuded oil was previously mechanically held and is expelled partly spontaneously, and partly due to struc-

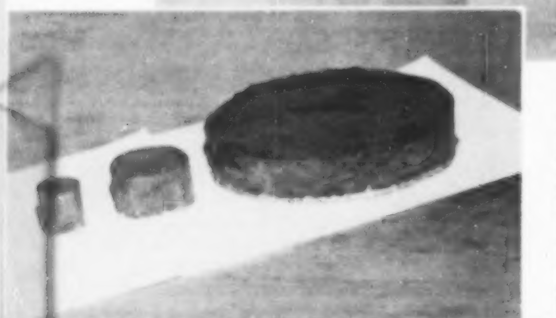
TABLE VI
DATA ON GREASES

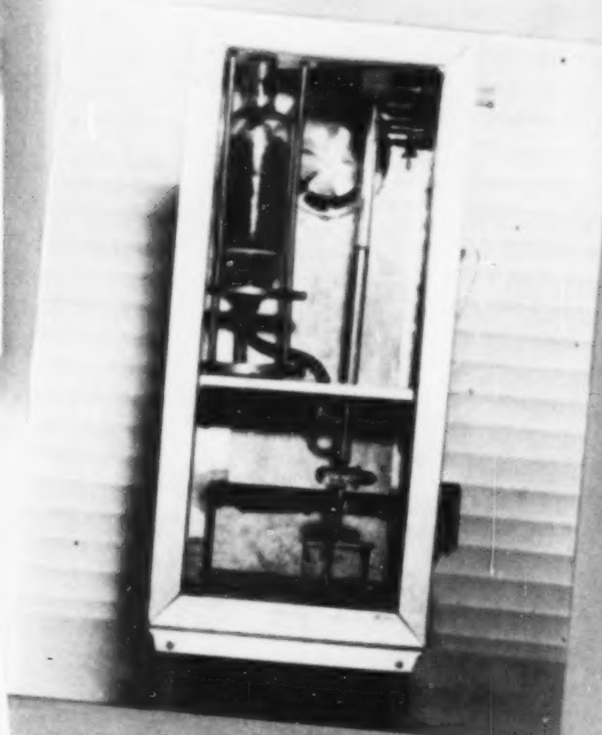
Number	Soap	Texture	Soap Content	A.S.T.M. Worked Penetration	Viscosity of Oil Component S.U.S. 100°F.	Viscosity Index of Oil Component
B1	Barium soap	short fiber	23.0	263	219	30
B2		short fiber	25.1	263	219	30
B3		short fiber	17.7	337	219	30
B4		short fiber	24.7	278	219	30
B5		short fiber	23.0	280	219	30
B6		short fiber	23.0	287	219	30
B7		short fiber	18.1	337	219	30
B8		short fiber	22.3	290	219	30
B9		short fiber	—	323	219	30
B10		short fiber	—	322	219	30
B11		short fiber	18.1	337	219	30
B12		short fiber	—	362	219	30
B13		short fiber	21.7	300	219	30
B14		short fiber	28.8	310	219	30
B15		short fiber	25.4	300	219	30
B16		short fiber	22.6	296	219	30
B17		short fiber	25.0	337	219	30
B18		long fiber	12.7	329	507	24
B19		long fiber	18.3	274	507	24
B20		buttery	17.7	338	450	57
B21		buttery	20.7	267	450	57
S1	Sodium soap	short fiber	18.0	282	1300	61
S2		short fiber	—	323	737	77
C1	Calcium soap	buttery	14.8	282	219	30
C2		buttery	17.04	237	219	30



Upper Right: Type of Container Used to Vary Static Head.

Lower Left: Type of Container Used to Vary Surface Area Volume Ratio.





Above: Pressure Stability Apparatus with Front Glass Cover Removed.

Upper Right: Crater Test Apparatus

Lower Left: Herschel Press, shown dismantled on the left, and assembled on the right.

tural changes in the gel framework. Increase in temperature, surface area, or static head would tend to accelerate these processes.

It would follow that the behavior of a grease under an applied pressure or stress would probably not be comparable with its behavior on ageing in storage. That this is correct has been demonstrated by the fact that the results of the Herschel, Centrifuge, and Pressure Filtration tests do not correlate with the results of the Crater Test, the latter being the only test which agrees with field storage behavior. Both pressure and filtration are employed in the Herschel and Pressure Filtration tests, and the results of these two tests were found to be in some degree comparable. However, they did not agree with the Centrifuge Test results, in which some other mechanism of syneresis is probably operative, possibly due to the larger applied force and the absence of a filtering membrane.

Controversy has arisen over the effect of syneresis on lubrication. Although a grease which breaks down rapidly and completely into an oil phase and a concentrated soap phase would have no value as a lubricant, a small amount of oil separation does not seem to impair lubricating efficiency. On the contrary it has been suggested that a small amount of syneresis is generally beneficial (5,17,34,38). For example, the presence of free oil ensures immediate lubrication in anti-friction bearings, possibly because of the wetting action on the bearing parts. No definite information on this question appears to have been published, and further investigations are necessary to clarify this situation.

SUMMARY

1. On storing most greases under static conditions, a small amount of oil tends to separate and collect on the surface.
2. The rate and amount of syneresis increases with temperature. It appears that the temperature dependence of syneresis is independent of soap content for any one type of grease.
3. The magnitude of syneresis is larger the lower the soap content, other conditions being held constant.
4. Increase in surface area causes an increase in syneresis.
5. Syneresis is also increased by increases in the static head of grease.
6. The Herschel Press, Pressure Filtration, and Centrifuge tests have been shown to be inadequate in predicting the syneretic behavior of a grease on storage.
7. A storage test designated as the "Crater Test" has been found to agree fairly well with storage behavior.
8. All the syneretic phenomena associated with greases can be explained by a simple mechanical hypothesis, which envisages the expulsion of oil from capillary spaces either spontaneously or due to the presence of internal stresses.

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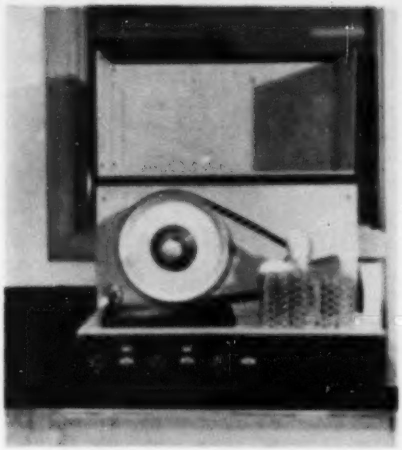

By C. W. GEORGI
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Evaluation OF WHEEL BEARING GREASES with the A.S.T.M. WHEEL BEARING GREASE TESTER

FOREWORD

The commonest source of complaint on the performance of wheel bearing greases is leakage from wheel hubs, especially in cases where sufficient leakage of grease occurs as to wet the brake bands and cause brake slippage. In connection with this problem, there is an all too prevalent tendency in the field to over-stuff wheel hubs with grease at the time of servicing, and such over-greasing is in many instances a major cause of leakage troubles. Another common cause of leakage difficulty is neglect of hub seals, wherein worn seals are not replaced, or the seal lips are damaged, and again not replaced, at the time a hub is dissembled for servicing.

Leakage tendencies from wheel hubs are thus dependent not only upon the grease, but also upon how much grease is applied, and upon the condition of the hub seals. In formulating and manufacturing a wheel bearing grease, it is accordingly necessary to "build in" not only leakage resistance per se, but also resistance to the adverse conditions of over-filling of hubs and defective hub seals. In similar fashion, to evaluate the leakage tendencies of wheel bearing greases it is necessary not only to study leakage per se, but also the effects of over-filling of hubs and of defective hub seals.

Considering the A.S.T.M. Wheel Bearing Grease Tester on this basis:

(1) It can be operated over a wide range of speeds and temperatures, so that the effects of centrifugal force, bearing "pumping", and heat may be adequately evaluated.

(2) It contains no seals, so that the worst condition likely to be encountered in service is incorporated in the tester.

(3) The hub of the tester may be filled with varying quantities of grease, so that the effects of over-filling can be studied.

Test work to date with the A.S.T.M. Wheel Bearing Grease Tester has been confined largely to test conditions following closely those given in the Proposed Method of Test, as published for information only in the 1948 Edition of A.S.T.M. Standards on Petroleum Products and Lubricants.

The 6-hour test period at 220° F. seems to be adequate insofar as the factors of temperature and time on grease leakage are concerned. Greases unsuited for wheel bearing service have been found to display high leakage results in well under 6 test hours. Similarly, the 220° F. test temperature has been found adequately high to detect unsuitable greases, yet not so high as to make the test unduly or unnecessarily severe. Test speeds equivalent to 40, 50 and 60 road miles per hour have been studied quite extensively, and it should not be difficult to determine which of these is most suitable.

However, in practically all of the cooperative work to date, the amount of grease applied to the hub has been arbitrarily set at 90 grams, so that the very important factor of over-filling and its relation to leakage has been neglected. The subject series of tests has therefore been directed to a more detailed study of test speed and grease sample charge, and their relation to leakage evaluation.

TESTS ON EIGHT GREASES OF KNOWN SERVICE PERFORMANCE

Eight greases, of fairly well defined and known service performance, were used in the initial studies to determine the effects of test speed and grease sample charge on leakage. Service experience with these greases was obtained from high speed passenger car road tests, controlled fleet tests in passenger cars and light trucks, and several uncontrolled fleet operations where grease performance was checked through cooperation of the fleet operators. Data on these greases are shown in Table 1.

TABLE 1
TEST GREASES

Grease No.	Leakage Rating In Service Performance	Penetration Unworked	Penetration 60 Strokes	Penetration 10,000 Strokes	ASTM Dropping Point
G-1	Good	190	216	300	325°
G-2	Good	206	248	328	350°
G-3	Good	280	275	320	370°
G-4	Good	260	283	310	370°
P-1	Borderline. OK On leakage when bearings carefully serviced. Leakage experienced with overfilled hubs.	233	267	357	300°
P-2	Poor	270	320		260°
P-3	Poor	255	260	315	over 400°
P-4	Poor	260	275	345	over 400°

TABLE 2
A.S.T.M. WHEEL BEARING MACHINE TESTS
6 Hours at 220°F.
450 RPM (40 m.p.h.)

LEAKAGE TO REAR COLLECTOR CUP

	90 gram Charge	150 gram Charge
G-1	Trace	8. grams
G-2	Trace	2. grams
G-3	Trace	13. grams
G-4	Trace	15. grams
P-1	1. grams	8. grams
P-2	3. grams	20. grams
P-3	trace	87. grams
P-4	trace	74. grams

TABLE 3
A.S.T.M. WHEEL BEARING MACHINE TESTS
6 Hours at 220°F.
560 RPM (50 m.p.h.)

LEAKAGE TO REAR COLLECTOR CUP

	90 gram Charge	110 gram Charge	130 gram Charge	150 gram Charge
G-1	trace	trace	8. grams	10. grams
G-2	trace	trace	1. grams	16. grams
G-3	trace	2. grams	4. grams	14. grams
G-4	1. gram	3. grams	6. grams	23. grams
P-1	1. gram	10. grams	34. grams	48. grams
P-2	7. gram	42. grams	60. grams	——
P-3	2. gram	40. grams	60. grams	——
P-4	4. gram	17. grams	58. grams	——

It will be noted there is no correlation between the service ratings of these greases and their respective penetrations or dropping points.

Table 2 shows results of tests on the 8 greases at 450 RPM (40 mi. per hr.) with grease sample charges of 90 grams and 150 grams (with 150 gram charge the hub is practically full).

With the 90 gram charge, there are no significant differences in leakage, and all 8 greases would be rated as satisfactory, which does *not* correlate with their service records. With the 150 gram charge at 40 mi. per hour test speed, greases P-3 and P-4 are rated as unsatisfactory, but greases P-1 and P-2 are still rated at a similar order of leakage as the "G" samples.

Table 3 shows results of tests at 560 RPM (50 mi. per hr.), and with varying grease sample charges.

With the 50 mi. per hour test speed, significant differences become evident, depending upon the sample charge. With the 90 gram charges, there is again little indicated difference between the 8 greases as to leakage. With the 110 gram charges, the "G" greases are all rated as superior to the "P" greases, but samples P-1 and P-4 are still rated better in leakage than their service records would indicate as correct. With the 130 gram charges, the "G" greases are all rated as markedly better than the "P" samples, and the test results are now in proper correlation with indicated service performance. With the 150 gram charges, the same relative order of rating prevails, although the leakage values are all appreciably higher, as might be expected with the test hub stuffed nearly full.

Two significant items are indicated by these tests at the 50 mi. per hr. speed:

(1) The "G" samples display only a gradual increase in leakage with increase of sample charge from 90 up to 150 grams, whereas the "P" samples display a marked increase in leakage with increasing sample charge. This sample charge—leakage relation indicates differences between greases which cannot be detected by test procedures using only an arbitrarily set 90 gram charge. Test results with 90 and 110 gram charges did not correlate with indicated service performance, while the tests with 130 and 150 gram charges are indicated to correlate quite satisfactorily.

(2) The 40 mile per hour test speed does not seem to exert sufficient centrifugal and bearing "pumping" forces to rate leakage tendencies adequately, and a higher test speed seems necessary. This may be due to dimensional conditions within the tester, or to the fact higher speed operation on the road may be more severe on greases, and that the tester is confirming this.

Table 4 shows results of tests at 680 RPM (60 mi. per hr.) with 130 and 150 gram charges.

The test results at the 60 mi. per hour test speed are substantially the same as those at 50 m.p.h., so that the speed factor can evidently be defined as too low at 40 m.p.h., and satisfactory at either 50 or 60 m.p.h.

HUB CAP LEAKAGE, GREASE "SLUMPING", STRUCTURE CHANGES and BEARING LUBRICATION

The present proposed test procedure calls for measurement of leakage to the hub cap, as well as notations on bearing lubrication, grease "slumping" from the hot hub and many structural changes in the grease after test. Table

5 lists typical data on this type from the tests on the 8 samples reported in the preceding.

Leakage to the hub cap was erratic in all of the tests, and as illustrated by Table 5, there was no correlation with indicated service performance. Similarly, there was no correlation between "slumping" and service record, and all 8 greases rated satisfactory as to bearing lubrication and resistance to structural changes. It appears the A.S.T.M. Wheel Bearing Grease Tester will serve best to evaluate grease leakage tendencies in terms of leakage to the rear collector cup, whereas other measurements or notations may have limited significance in relation to service performance.

TESTS ON 20 COMMERCIAL WHEEL BEARING GREASES

As a matter of general interest, 20 samples of commercial wheel bearing greases were tested by modified procedures. Based upon the test results with the first 8 grease samples, the 50 mi. per hour test speed with 130 gram grease charge was selected.

As shown, there is no sharp dividing line between greases having "good" leakage ratings and those having "poor" ratings. In order to secure a sharper differentiation, further tests were run on the "borderline" greases, using a 150 gram charge, with results as summarized in Table 7.

These results show the same condition experienced with the first 8 test greases, in that some products display very little difference in leakage in tests with either 130 or 150 gram charges, whereas other greases develop a sharp increase in leakage in going from a 130 gram to a 150 gram charge.

Based upon the test evidence accumulated, it appears greases developing leakage of less than about 15 grams with 130 gram charge may be rated as "good". With leakage over 15 grams and up to about 30 grams, the rating may be considered as "borderline", and above 30 grams as "poor". For added evaluation, greases rated as "borderline" may be retested with 150 gram charge; those showing only slightly increased leakage and below about 25 to 30 grams then being considered as satisfactory, and those showing markedly increased leakage (above about 30 grams) then being given a final rating of "poor".

As in previous tests, leakage to the hub cap and "slumping" from the hot hub was very erratic and bore no relation to the rear cup leakage ratings of the respective greases. Bearing lubrication was satisfactory and there were no significant grease structural changes after test with all 20 samples.

The above values suggested for this method of leakage rating are based upon work in only one laboratory, so they cannot be proposed as final or conclusive figures. Leakage values by the modified procedure may vary considerably in other laboratories due to differences in operating and hub packing technique, or to apparatus variations not yet defined. Generally acceptable leakage values for tests with grease sample charges greater than 90 grams cannot be developed until cooperative studies are undertaken to evaluate and standardize the modified test procedure.

Certain modifications of the test machine may also be desirable. A larger rear collector cup would be desirable, since the present cup was not designed for catching the higher amounts of leakage developed in many cases with larger grease sample charges. Also, the larger charges may well re-

TABLE 4

A.S.T.M. WHEEL BEARING MACHINE TESTS

6 Hours at 220°F.
680 RPM (60 m.p.h.)

	LEAKAGE TO REAR COLLECTOR CUP	
	130 gram Charge	150 gram Charge
G-1	9. grams	15. grams
G-2	8. grams	22. grams
G-3	7. grams	28. grams
G-4	9. grams	20. grams
P-1	33. grams	48. grams
P-2	58. grams	——
P-3	——	——
P-4	——	——

TABLE 5

A.S.T.M. WHEEL BEARING MACHINE TESTS

6 Hours at 220°F. 560 RPM (50 mi. per hour)
130 gram Sample Charge

	Leak. to Hub Cap	"Slumping" From Hot Hub	Structural Changes In Grease after Test	Bear. Lub.
G-1	nil	No	No significant structural change	Okay
G-2	6.	No	No significant structural change	Okay
G-3	7.	No	No significant structural change	Okay
G-4	nil	No	No significant structural change	Okay
P-1	6.	No	No significant structural change	Okay
P-2	nil	Yes	No significant structural change	Okay
P-3	1.	No	No significant structural change	Okay
P-4	8.	Yes	No significant structural change	Okay

TABLE 6

TESTS ON 20 COMMERCIAL WHEEL BEARING GREASES

Test Conditions: 6 Hrs. at 220°F.
Speed: 560 RPM (50 m.p.h.)
130 gram Grease Charge

Leakage to Rear Collector Cup	Leakage Rating	Number of Greases
Below 10 grams	Good	8
10 to 20 grams	Good to Borderline	4
20 to 30 grams	Borderline to Poor	2
Over 30 grams	Poor	6

quire a carefully standardized procedure for packing the hub.

TEST REPRODUCIBILITY

Test reproducibility with larger grease charges is indicated by typical check tests listed in Table 8.

Although test reproducibility is not highly precise with some greases, the reproducibility is indicated to be adequate for differentiating between products having "good", "borderline" and "poor" leakage ratings.

SUMMARY

1. The commonest source of complaint on the performance of wheel bearing greases is leakage from wheel hubs. There is an all too prevalent tendency in the field to over-stuff wheel hubs with grease at the time of servicing, and such over-greasing greatly aggravates leakage tendencies and troubles.

2. In all of the cooperative work to date with the A.S.T.M. Wheel Bearing Grease Tester, the amount of grease applied to the hub has been arbitrarily set at 90 grams, so that the very important factor of over-filling and its relation to leakage has been neglected.

3. The test speed of 450 R.P.M. (40 mi. per hr.) does not seem to exert sufficient centrifugal forces to rate grease leakage tendencies adequately. Test speeds of 560 and 680 R.P.M. (50 and 60 mi. per hr. respectively) indicate better correlation with service performance. Test results at the 60 mi. per hr. test speed were substantially the same as those at 50 mi. per hr., so that the speed factor can evidently be defined as too low at 40 mi. per hr., and satisfactory at either 50 or 60 mi. per hr.

4. Leakage test results with 90 and 110 gram sample charges did not correlate with indicated service performance, while tests with 130 and 150 gram charges are indicated to correlate quite satisfactorily.

5. Based upon the test evidence accumulated, it appears a modified test procedure will evaluate the leakage tendencies of wheel bearing greases in satisfactory accord with their service performance:

6 hours at 220° F.
560 RPM (50 mi. per hr.)
130 and 150 gram sample charges.

By this test procedure, greases developing leakage of less than about 15 grams with 130 gram sample charge may be rated as "good". With leakage over 15 grams and up to about 30 grams, the rating may be considered as "borderline"; and above 30 grams as "poor". Greases rated as "borderline" may be retested with 150 gram charge; those showing only slightly increased leakage and below 30 grams then being rated as satisfactory; and those showing markedly increased leakage (above 30 grams) then being given a final rating of "poor".

The above values suggested for this method of leakage rating are based upon work in only one laboratory, so they cannot be proposed as final or conclusive figures. Generally acceptable leakage values for tests with grease sample charges greater than 90 grams cannot be developed until cooperative studies are undertaken to evaluate and standardize the modified test procedure. Certain modifications of the test machine and of details of test technique may also be desirable.

6. Reproducibility of the modified test procedure is indicated to be adequate for the intended purpose of evaluating the comparative leakage tendencies of wheel bearing greases.

7. It appears the A.S.T.M. Wheel Bearing Grease Tester serves best to evaluate the leakage tendencies of greases in terms of leakage to the rear collector cup. Other measurements and notations such as leakage to the hub cap, grease "slumping" from the hot hub, bearing lubrication, and structural changes in the grease after test, appear to have only limited significance in relation to service performance.

TABLE 7

TESTS ON COMMERCIAL WHEEL BEARING GREASES HAVING "BORDERLINE" LEAKAGE RATINGS

(6 Hours at 220°F., 560 RPM)

	Leakage to Rear Collector Ring		
	130 Gram Charge	150 Gram Charge	Final Leakage Rating
#A	16. gms.	17. gms.	Good
#B	16. gms.	19. gms.	Good
#C	15. gms.	37. gms.	Poor
#D	19. gms.	53. gms.	Poor
#E	25. gms.	43. gms.	Poor
#F	30. gms.	60. gms.	Poor
#G	34. gms.	48. gms.	Poor
#H	34. gms.	60. gms.	Poor

TABLE 8

CHECK TESTS

A.S.T.M. WHEEL BEARING GREASE TESTER

6 Hours at 220° F. 560 RPM (50 mph)

130 gram Grease Charge

Grease	Leakage to Rear Collector Cup (grams)			Leakage to Hub Cap (grams)		
P	1.0	1.2		5.9	8.5	
Q	2.2	1.2		nil	nil	
R	9.8	15.9	8.9	1.1	4.1	nil
S	16.1	9.1	11.9	nil	4.2	nil
T	19.0	28.5	26.6	17.0	8.5	17.6
U	24.7	21.2		1.5	.6	
V	34.1	56.3		3.6	11.8	
W	47.8	52.5		7.4	2.7	
X	54.8	63.0		3.0	3.7	

C. W. GEORGI PUBLISHES NEW LUBRICATION BOOK

Past president, Carl W. Georgi, author of the new book "Motor Oils and Engine Lubrication", is widely known in the automotive and petroleum industries for a large number of papers and articles published on subjects pertaining to lubricants and lubrication.

In addition to his duties as technical director at the research laboratories of the Quaker State Oil Refining Corporation and Vice-President of the Enterprise Oil Co., Inc., he plays an active part on technical committees which deal with lubricants and lubrication in such societies as the Society of Automotive Engineers, American Petroluem Institute, American Society for Testing Materials, Co-ordinating Research Council, National Lubricating Grease Institute, American Chemical Society, and American Society of Lubrication Engineers.

"YOUR EXCUSE FOR NOT TAKING ME WITH YOU TO N.L.G.I. MEETINGS IN THE PAST HAS BEEN 'LACK OF HOTEL SPACE'—WHAT IS IT **THIS** YEAR, GEORGE?"

IT WILL BE **WORTH** THE EXPENSE, MISTER!

FUTURE MEETINGS OF YOUR INDUSTRY

JULY

3-7 The Institute of Petroleum (2nd oil shale and cannel coal conference), Glasgow, Scotland

12-14 Institute of the Aeronautical Sciences (annual summer meeting), I. A. S. Western Headquarters Building, Los Angeles, Calif.

AUGUST

1-4 National Congress of Petroleum Retailers (national convention), Washington Hotel, Seattle, Wash.

3-5 Interstate Oil Compact Commission, (summer meeting), French Lick Springs Hotel, French Lick, Ind.

14-16 Socy. of Automotive Engineers (west coast meeting) Biltmore Hotel, Los Angeles, Calif.

15-18 Delaware Firemen's Short Course, University of Delaware, Newark, Delaware

22 Oil Trades Assn. of New York, Pelham Country Club, Pelham, N. Y.

We'll See You At The N.L.G.I. Meeting - Edgewater Beach Hotel
October 30, 31 and November 1. Chicago, Illinois
Some Reservations Still Open

SEPTEMBER

3-8 American Chemical Society Chicago, Ill.

5-9 Sixth National Chemical Exposition, Coliseum, Chicago, Ill.

8-9 Michigan Petroleum Assn., (fall convention), Grand Hotel, Mackinac Island, Michigan.

10-13 American Inst. of Chemical Engineers (regional meeting), Radisson Hotel, Minneapolis, Minn.

11-13 Oil Industry Information committee, Traymore Hotel, Atlantic City, N. J.

12-14 Socy. of Automotive Engineers, (tractor meeting), Hotel Schroeder Milwaukee, Wis.

13-15 National Assn. of Motor Bus Operators (21st annual meeting, Drake Hotel, Chicago, Ill.

13-15 National Petroleum Assn., Hotel Traymore, Atlantic City, N. J.

14 American Petroleum Institute, (Lubrication Committee), Hotel Traymore, Atlantic City, N. J.

18-22 Fifth National Instrument Conference and Exhibit, Memorial Auditorium, Buffalo, N. Y.

19-23 American Socy. of Mechanical Engineers Hotel Sheraton, Worchester, Mass.

20-21 Ohio Petroleum Marketers Assn., (fall conference), Netherland Plaza Hotel, Cincinnati, Ohio

25-27 American Socy. of Mechanical Engineers (Petroleum Mechanical Engineering division) The Roosevelt, New Orleans, La.

25-27 American Trade Assn. Executives Somerset Hotel, Boston, Mass.

26-29 Iron and Steel Exposition and annual Convention of Iron and Steel Engineers, Cleveland Public Auditorium, Cleveland, Ohio

27-29 National Metal Trades Assn. Hotel Commodore, New York, N. Y.

27-30 Socy. of Automotive Engineers (aeronautic meeting and aircraft engineering display) Biltmore Hotel, Los Angeles, Calif.

OCTOBER

1-3 Independent Petroleum Assn. of America (annual meeting) Jefferson Hotel, St. Louis, Mo.

3-5 American Inst. of Electrical Engineers (district No. 2), Lord Baltimore Hotel, Baltimore, Md.

11 American Iron and Steel Inst. (regional technical meeting), Hotel William Penn., Pittsburg, Pa.

12-13 Indiana Independent Petroleum Assn. (fall convention) Hotel Severin, Indianapolis, Ind.

16-18 Socy. of Automotive Engineers (transportation meeting) Hotel Statler, New York, N. Y.

16-20 National Safety Congress Chicago, Ill.

16-21 Oil Progress Week

19-22 Permian Basin Oil Show, Odessa, Texas

20-21 American Management Assn., Hotel Statler, New York, N. Y.

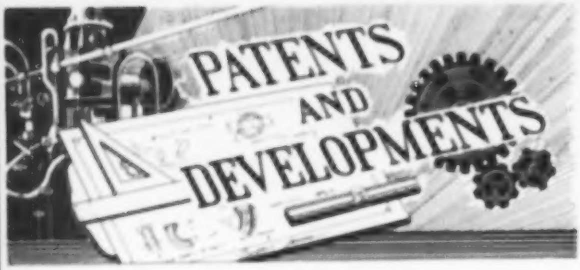
23-27 American Inst. of Electrical Engineers (fall general meeting), Skirvin Hotel, Oklahoma City, Okla.

23-27 National Metal Exposition Amphitheatre, Chicago, Ill.

24-25 South Dakota Independent Oil Men's Assn. Aberdeen Civic Arena, Aberdeen, S. D.

25 American Iron and Steel Inst. (regional technical meeting), Hotel Thomas Jefferson, Birmingham, Ala.

"I GUESS THIS MEANS MY WIFE HAS DECIDED TO ACCOMPANY ME TO THE N.L.G.I. ANNUAL MEETING IN CHICAGO!"



STABILIZED GREASES—One patent issued to Gulf Oil Corp. discloses production of greases of increased stability against oil separation and structural breakdown at high and low temperatures, and having improved resistance to oxidation.

The stabilizer used is a neutral higher fatty mono-carboxylic acid ester of a fusible, soluble mono-alkylated phenol-formaldehyde condensation product, the higher fatty acid being a saturated or monoolefinic acid having at least 8 carbon atoms, while the alkyl substituent on the alkylated phenol has 4 to 12 carbon atoms. These esters are prepared by esterification of the alkyl phenol-formaldehyde condensation product at a temperature of not over 500° F. The fatty acid is used in practically equimolar amounts with the alkyl phenol.

It is preferred to employ diisobutylene for the alkyl group on the phenol since the alkyl phenol obtained is primarily para—(alpha, alpha, gamma, gamma) tetramethylbutyl phenol which is particularly useful in preparing grease additives. However, n-alkyl phenols can be employed. As to the fatty acid for esterification of the hydroxy groups in the condensation product, the following may be used: caprylic, pelargonic, capric, undecylic, lauric, myristic, palmitic, stearic, arachidic, behenic, carnaubic, cerotic, melissic, psyllaic, nonylenic, decylenic, undecylenic, oleic, ricinoleic, erucic and brassidic.

This ester product is a brownish waxy material resembling beeswax in odor and appearance, and has the following properties:

Specific gravity (solid)	1.11
Melting point, °F	108-20
Neutralization number	8.6

Greases prepared with such stabilizer esters have withstood oxidation at 176°F for over 500 hrs., in comparison with unstablized greases which failed at 20 hrs. Also, oil separation was practically nil with such stabilized greases. About 0.1 to 5% of the stablizer is employed (U.S. 2,506,905.)

TEXTURE-STABLE GREASE COMPOSITION—Development of lubricating greases particularly designed for specialty equipment employing shielded or sealed bearings and operating under wide temperature ranges has been described in a patent issued to the Texas Co. These greases contain silicones which offer a marked advantage over mineral oils in that they possess a comparatively uniform viscosity over a wide temperature range and are not as susceptible to evaporation loss as a comparable mineral oil. Synthetic silicone greases heretofore available have one undesirable characteristic, which is lack of resistance to shear. Under high shearing stresses such as in ball or roller lubrication, the structure breaks down and their consistency falls off until they become liquid or semi-liquid. The present compositions are claimed to overcome these characteristics by the use of special acid components of the metal soap, namely soapforming hydroxy fatty acids or their glycerides.

The silicone polymers which may be used are the butyl, amyl or higher although the preferred materials are the dimethyl, diethyl, ethylphenyl or methylphenyl silicone polymers which may be the sole oil component of the grease or may be blended with mineral oil if desired.

The soap-forming hydroxy fatty acids are those containing at least 12 carbon atoms in the molecule and one or more hydroxyl groups separated from the hydroxyl group by at least 1 carbon atom. These may be obtained from caster oil, oxidation of unsaturated fatty acids or oils and waxes, 12 hydroxy stearic acid, or hydrogenated castor oil.

The metallic constituent of the soaps may be any of the metals such as sodium, lithium, calcium, barium, aluminum, etc. A typical product obtained is a smooth, light-colored, buttery grease having the following calculated composition:

Lithium soap	12.8%
Glycerine	0.9%
Mineral oil	13.4%
Dimethyl silicone polymer	72.9%

Such a grease possesses a fairly high dropping point, is low in evaporation loss and low in oil bleeding and also meets torque requirements at -67° F. In addition, its grease break-down test performance is outstanding since it shows the product to behave excellently up to 300° F without break-down or change in texture or body (U.S. 2,508,741).

ANHYDROUS BARIUM SOAP GREASES—A method of preparing an anhydrous normal barium soap grease having good water and heat resistant properties and stable against oleaginous liquid and soap separation, involves saponifying, in the presence of an oleaginous liquid of lubricating viscosity constituting a fractional part of the oleaginous liquid used in the final grease, and acidic soap-forming material containing predominant proportion of soap-forming hydroxy fatty acids, with barium hydrate at temperatures of 170-200° F to produce a saponified product of normal barium soap, heating this saponified product to effect substantially complete dehydration at temperatures above 260° F but below 300° F, adding additional oleaginous liquid of lubricating viscosity in substantial amount to obtain a stiff adhesive grease consistency while cooling the dehydrated product to a temperature below 200° F, all of these operations being conducted with continuous stirring, and finally drawing the resulting anhydrous grease composition at a temperature below 200° F (U.S. 2,503,749).

HIGH TEMPERATURE LUBRICATING GREASES—One lubricating grease composition for high temperature use recently patented by Standard Oil Development Co. consists essentially of a mineral base lubricating oil containing 20-40% by weight of sodium soap of rapeseed oil, the composition having a substantial glycerine content and a content of 0.25-2% excess alkali calculated as free NaOH, 0.1-2% phenyl alpha napthylamine, and 0.1-1% of dibenzoyl acetone alkylene diamine in which the alkylene group is ethylene or propylene (U.S. 2,503,969).

GREASE—Among the improved lubricating grease compositions disclosed in a Canadian patent by Standard Oil Development Co. is one containing a low pour point lubricating oil distillate together with 10-25% of alkali soap such as the sodium soap of erucic acid or acids derived from rapeseed oil, a substantial portion of which soap is a soap of a monocarboxylic aliphatic acid of 22 carbon atoms, and a soap of an amphoteric metal with acid having an iodine number under 30, such as 0.5% aluminum stearate and 0.5% zinc napthenate (Can. 464,221).

HAND-OPERATED GREASE GUN—K-P Mfg. Co. is marketing a utility grease gun which can be operated with one hand and claimed to eliminate air pockets (Nat'l Petr. News 5/17/50 p.49).

CHOICE OF GREASE MAKING EQUIPMENT—In a recent article, Boner of Battenfeld Grease & Oil Corp., summarizes the best practices and most efficient equipment developed for the lubricating grease manufactuuring industry (Petr. Engr. 5/50 C7).

GREASELESS LUBRICANT—Warren Refining & Chemical Co. is marketing Plastilube, a grease containing montmorillonite reacted with organic cations (Chem. Ind. 5/50 p. 724).

PLUG VALVE LUBRICATOR—A new automatic plug valve lubricator, made by Delta Engrg. & Sales Co. forces a measured amount of lubricant into the lubricating channels of the valve as the valve is opened and closed (Chem. Ind. 5/50 p. 742).

GREASES HEAVIER THAN WATER—British & U.S. Navy Submarine Services have worked on the problem of the oil slick which often gives away the presence of submarines and have developed a calcium base grease loaded with finely dispersed powdered metallic lead, the gravity of the lubricant being greater than that of sea water (Lead, Vol. 18, No. 1, 1950 p.5).

NON-MELTING GREASES—McCarthy discloses non-melting Bentone greases containing montmorillonite compounds (Abstracts 24th Nat'l Colloidal Symposium, St. Louis 6/50 p.12).

SOAP-OIL-WATER DISPERSIONS—Transitions in soap-oil-water systems by dielectric loss measurements are discussed by Doscher (Abstracts 24th Nat'l Colloidal Symposium p.15).

PATENTS AND APPLICATIONS—

U. S.

2,503,442 (The Texas Co.)—Grease lubricated journal bearing with deflectors.

2,504,055 (Stewart-Warner Corp.)—High pressure grease fitting.

2,506,204 (Freestone)—Grease dispenser.

2,509,863 (Standard Oil Development Co.) — Divalent metal sulfonates.

CAN.

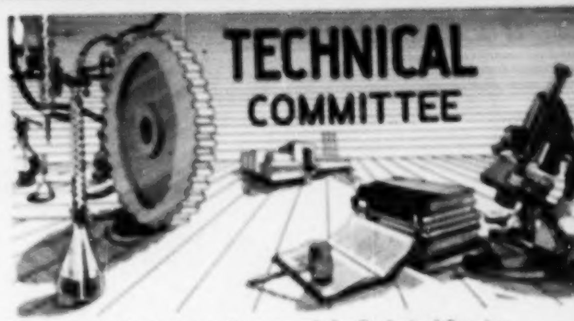
465,359 (Girdler Corp.) — Continuous production of greases (U.S. patent previously discussed.)

BRIT. PAT.

639,203 (Shell Oil)—Anhydrous sodium soap lubricating greases.

BRIT. APPL.

24,741/49 (Standard Oil Development Co.) — Lubricating grease compositions.



Chairman T. G. Roehner, Director of the Technical Service Department, Socony-Vacuum Laboratories

ABEC-NLGI Cooperative Committee on Grease Test Methods held its Annual Meeting at the ABEC Headquarters on June 9. A summary of the minutes of that meeting will be given herein as soon as they have been approved. In the meantime, it may be of interest to comment briefly on the subject that received the most attention, namely, the revised AFBMA Guide for the Selection of Lubricants for Ball and Roller Bearings, which will be released for general distribution in the near future. The BEC Tester has been redesigned and arrangements are under way to again have it manufactured by one or more companies. The new machine is an important improvement over the original design, and it is planned to offer the unit to ASTM Technical Committee G to be included with other testers they have under consideration for functional tests of ball bearing greases. It will be recalled that ASTM Technical Committee G heretofore has focused most of its effort on the so-called Navy and GE Machines. The BEC Tester will differ in that it is primarily a screening device. The other two testers are built for comparatively long-time runs until failures of the greases or bearings have occurred.

At the February Meeting of ASTM Technical Committee G, it was decided that a group would be organized under the chairmanship of Marshall Anderson to follow all developments connected with the GE Ball Bearing Grease Tester. A proposed method was distributed covering the recommended procedure to be used with that equipment. This step was taken in recognition of the fact that there are about twenty of those machines in service and that each laboratory apparently follows a different procedure. It was hoped that thru this group the laboratories would be led to adoption of more uniform operating conditions so that pooling of their experience could be made more effective. The membership of this particular group will be limited to members who are now using that tester, and it is hoped that within the next year they will be in a position to report sufficient information on which to base a decision regarding the publication of the method by ASTM on an "information only" basis. It may be of interest to recall that this particular tester was designed in accordance with specifications proposed by a Subsection of ASTM Technical Committee G, and essentially it is intended to enable reasonable duplication of the operating conditions encountered in conventional electric motors fitted with ball bearings. The type of bearings and loading are comparable to those expected under actual service conditions. Moreover, the heat source is so located that the temperature of the grease will rise in about the same manner as in an actual electric motor.

THE N.L.G.I GUIDE POST

Industry CROSSROADS FOR BUYERS and SELLERS

SUPPLIERS OF MATERIALS FOR MANFACTURING LUBRICATING GREASES

MANUFACTURERS OF EQUIPMENT FOR APPLICATION OF LUBRICATING GREASES

A.S.T.M. AWARDS FOR NOTABLE TECHNICAL PAPERS

In recognition of outstanding technical papers presented at its previous Annual Meetings, the American Society for Testing Materials made awards for such three papers, the authors receiving the awards at the Tuesday evening session of 1950 Annual Meeting in Atlantic City, during the week of June 26. The awards were as follows:

CHARLES B. DUDLEY MEDAL—to Professor B. J. Lazan, Syracuse University, Syracuse, N. Y., for his paper entitled "Dynamic Creep and Creep-Rupture Properties of Temperature—Resistant Materials under Tensile Fatigue Loading." This medal is presented for a paper of outstanding merit constituting an original contribution on research in engineering materials.

RICHARD L. TEMPLIN AWARD—to Professors D. S. Clark and D. S. Wood, California Institute of Technology, Pasadena, Calif., for their paper entitled "The Time Delay for the Initiation of Plastic Deformation at Rapidly Applied Constant Stress." This award is presented for a significant paper describing new testing methods and apparatus, the purpose of the award being to stimulate research in the development of testing methods and apparatus.

SAM TOUR AWARD—to O. B. Ellis, Senior Research Engineer, Armco Steel Corp., Middletown, Ohio, for his paper entitled "Effect of Weather on the Initial Corrosion Rate of Sheet Zinc." This award is presented for the purpose of encouraging research on the improvements and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field.

HI-SPEED CENTRIFUGE

A new high speed centrifuge which enables safe, rapid completion of the most difficult separations has been developed by Precision Scientific Company.

The 5000 r.p.m. Centrifuge was designed for semi-micro and micro qualitive analysis in all industries.

The six tube head is totally enclosed with each tube in a separate compartment. This is a valued feature when centrifuging volatile such as ether which would be evaporated by the air friction heat at high speeds. The head is finished in Neutragreen Hammerloid, a tough baked enamel resistant to attacks by acids, alkalies and temperature extremes.

A rheostat control provides speed control from 1800 to the maximum 5000 in unbroken sequence. Developing 1/20 H.P., the series wound motor operates on standard 115 volts A.C. to deliver 5000 r.p.m.—up to 20% high speeds on D.C. A reference dial permits the duplication of selected speeds. For fast stopping, a friction leather brake has been incorporated.

An all stainless steel housing encloses the entire mechanism including the access hole, which is covered by a stainless steel presure-fit perforated cap. The exterior is smoothly finished and brought to a high polish. "Walking" of the centrifuge is prevented by four suction cup feet.

Overall Dimensions: 12" in diameter by 8½" high.

AMERICAN SOCIETY FOR TESTING MATERIALS ELECTS SIX HONORARY MEMBERS

In recognition of their eminence in technical work in engineering materials, and for especially meritorious service to the organization over many years, the American Society for Testing Materials signally honored six of its prominent members by awarding them Certificates of Honorary Membership at its Annual Meeting luncheon session in Atlantic City on Tuesday, June 27. The men honored:

Phaon H. Bates, formerly of Washington, D.C., now St. Petersburg, Fla.

Wilson C. Hanna, Colton, Calif.

Dean Harvey, Pittsburgh, Pa.

Prevost Hubbard, New York City, N. Y.

Robert Job, Montreal, Canada.

Henry S. Rawdon, Wasington, D. C.

have had distinguished careers in various fields, but have done particularly notable work in various phases of A.S.T.M. work involving standardization and research in materials. Presentation was made by the Society's President, James G. Morrow. (Mr. Hubbard was unable to get to the meeting because of an important prior West Coast engagement, and Mr. Job could not be present.)

A.S.T.M. NOMINATES NEW OFFICERS

Nominations for new officers of the American Society for Testing Materials are announced. Official notice of election will be given at the 53rd A.S.T.M. Annual Meeting in Atlantic City, N. J., week of June 26.

PRESIDENT (Term—one year)

L. J. Markwardt

Assistant Director

U. S. Forest Products Laboratory

A.S.T.M. NOMINATIONS (cont.)

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Votator

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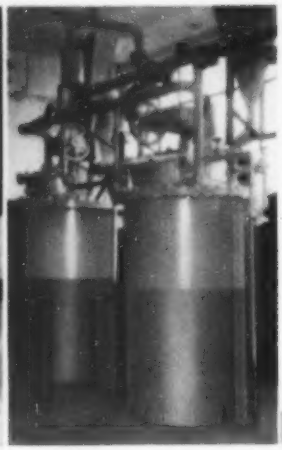

★ Aluminum and lithium stearate grease processing costs are lower with VOTATOR Grease Making Apparatus.

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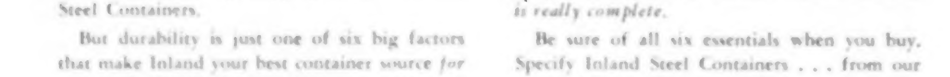
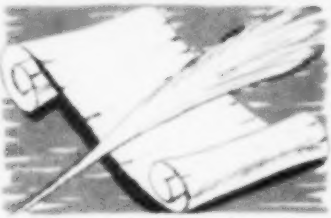


Associate and Technical Members . . . of the National Lubricating Grease Institute



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Tulsa, Oklahoma
Member—T. E. Fitzgerald

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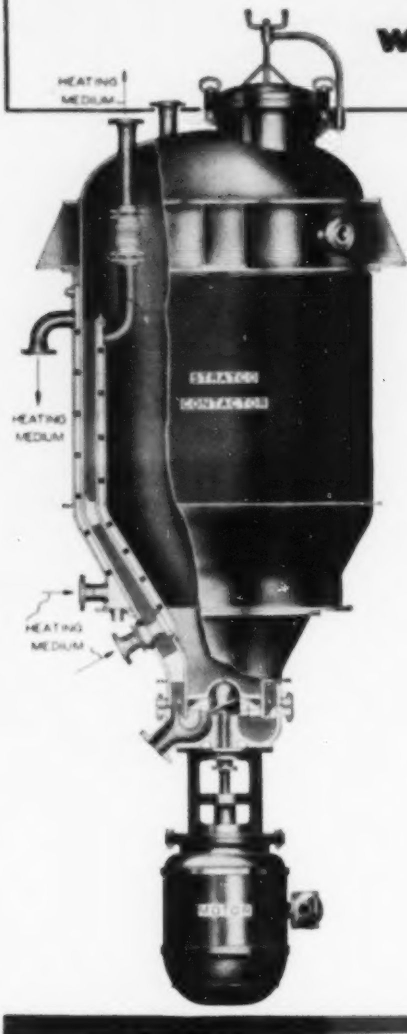
Kansas City 2, Missouri

GREASE MANUFACTURE SIMPLIFIED
Time Saved . . . Cost Reduced

with the

STRATCO



High Dispersion CONTACTOR

and Oil Circulation Heating System

This equipment puts your plant on an eight hour basis, simplifies laboratory control, reduces manpower requirements, produces a more uniform product at lower cost.

The Contactor is also adaptable to any other mixing problem where one or more of the materials to be mixed is a liquid.

STRATFORD ENGINEERING

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